factor $R=\sum||F_o|-|F_c||/\sum|F_o|$ decreased to 0·065. After a further 10 cycles of refinement of the full three-dimensional data R took a minimum value of 0·071, at which stage the shifts in the atomic parameters were all less than $\frac{1}{50}$ of the estimated standard deviations. In these calculations the atomic scattering factors given in *International Tables for X-ray Crystallography* (1962) for Cs^+, Br^- and Mn^{2+} were used and inter-layer scaling was achieved by scaling the observed to the calculated structure factors.

The calculated structure factors of 55 symmetrically independent unobserved reflexions were all less than the minimum observable value. The final atomic parameters are given in Table 2 and the observed and calculated structure factors compared in Table 3.

Table 2. *Final atomic parameters*

Standard deviations are given in parentheses.

	Equipoint	x	y	z	B(Å^2)
Mn	2*a*	0	0	0	1·78 (32)
Cs	2*d*	$\frac{1}{3}$	$\frac{2}{3}$	$\frac{3}{4}$	2·34 (17)
Br	6*h*	0·1617 (10)	0·3234 (10)	$\frac{1}{4}$	1·72 (11)

Table 3. *Observed and calculated structure factors*

H	K	F_o	F_c	H	K	F_o	F_c	H	K	F_o	F_c	H	K	F_o	F_o	H	K	F_o	F_c	H	K	F_o	F_c
L=0				6	2	29	-30	3	1	17	-24	2	2	151	-154	4	1	13	-11	6	1	16	15
2	0	96	-92	8	2	50	56	4	1	17	13	3	2	19	16	3	2	20	-20	2	2	173	158
3	0	69	60	3	3	39	38	3	2	32	25	4	2	86	82	4	2	108	100	3	2	26	23
4	0	70	-64	5	3	12	10	4	2	126	-117	6	2	52	52	5	2	17	16	4	2	35	-36
6	0	132	134	6	3	18	17	5	2	19	-19	7	2	27	24	6	2	66	-63	5	2	22	18
7	0	27	26	4	4	109	106	6	2	82	73	8	2	41	-38	6	4	40	41	6	2	20	-23
8	0	28	-24	5	4	17	21	6	4	55	-48	4	3	35	34	8	4	24	-23	8	2	39	43
1	1	81	75	6	4	22	-19	8	4	30	26	4	4	75	-74	L=4				3	3	20	26
2	1	7	10	6	6	35	38	L=2				6	4	35	34	2	0	58	-58	4	4	83	80
3	1	26	22	L=1				2	0	145	147	L=3				3	0	37	37	L=5			
4	1	48	43	2	0	188	-219	4	0	106	106	2	0	175	170	4	0	43	-45	2	0	114	-118
6	1	18	19	3	0	18	-18	5	0	43	43	3	0	17	14	6	0	97	99	4	0	94	93
7	1	19	21	4	0	149	153	6	0	89	-93	4	0	129	-128	7	0	13	20	8	0	37	-39
2	2	202	231	7	0	21	15	8	0	39	42	8	0	45	51	1	1	42	44	2	4	80	74
3	2	32	31	3	0	60	-59	2	1	55	53	10	0	23	-28	2	1	11	6	2	6	50	-48
4	2	53	-49	10	0	32	31	3	1	34	33	2	1	11	-14	3	1	20	15	4	6	30	32
5	2	30	26	2	1	17	19	5	1	29	30	3	1	14	19	4	1	26	29				

Discussion

Cs and Br atoms form an approximate hexagonal close-packed array such that each Cs atom has 12 nearest Br neighbours. The Mn atoms occupy those octahedral sites which are bounded exclusively by Br atoms.

The Mn–Br octahedra share opposite faces to form chains, of composition $(MnBr_3)_n^{n-}$, parallel to the c axis. The trigonal distortion of the octahedra is presumably due to the repulsion of successive Mn^{2+} ions in the chains so that the Br atoms in the shared faces are brought closer together, thereby making the Br(1)–Mn–Br(1) and Br(2)–Mn–Br(2) angles less than 90° and the Br(1)–Mn–Br(2) angle correspondingly greater. Bond lengths and angles are given in Table 4.

Table 4. *Bond lengths and angles*

Standard deviations are given in parentheses.

	Multiplicity	
Mn–Br octahedron:		
Mn–Br(1,2)	6	2·683 (6) Å
Br(1)–Br(1)	3	3·691 (12)
Br(2)–Br(2)	3	
Br(1)–Br(2)	6	3·893 (5)
Angles		
Br(1)–Mn–Br(1)	3	86·9 (0·2)°
Br(2)–Mn–Br(2)	3	
Br(1)–Mn–Br(2)	6	93·1 (0.2)
Cs–Br distances:		
Cs–Br(2)	6	3·805 (8) Å
Cs–Br(1)	3	3·967 (4)
Cs–Br(3)	3	

Location of atoms:

Mn at (000)

Cs at $(\frac{2}{3}\frac{1}{3}\frac{1}{4})$

Br(1), Br(2), Br(3) at $z=-\frac{1}{4}$, $\frac{1}{4}$ and $\frac{3}{4}$ respectively.

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The crystal structure of the nitroxide free radical 2,2,5,5-tetramethyl-3-carbamidopyrroline-1-oxyl.

By JUNE W. TURLEY and F. PETER BOER, *The Dow Chemical Company, Midland, Michigan* 48640, *U.S.A.*

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The crystal and molecular structure of 2,2,5,5-tetramethyl-3-carbamidopyrroline-1-oxyl, $C_9H_{15}N_2O_2$, has been determined by a three-dimensional X-ray diffraction study. The crystals are in the monoclinic space group $P2_1/c$, with four molecules in a unit cell of dimensions: $a=8·039$ (2), $b=11·324$ (2), $c=11·537$ (2) Å, and $\beta=91·75$ (1)° measured at 24°C; $D_x=1·160$ g.cm^{-3}. Intensity data were measured on a four-circle diffractometer using Cu $K\alpha$ radiation (1·5418 Å), and the structure was solved by statistical methods. Full-matrix least-squares refinement converged at $R=5·2$% for 1474 reflections above background, with anisotropic thermal parameters assumed for C, N, and O and isotropic thermal parameters for H. The ring system and the nitroxide function were found to be planar.

The current interest in nitroxide free radicals stems largely from their utility in electron spin resonance spectroscopy as spin labels in normally diamagnetic systems (Griffith & Waggoner, 1969; Snipes & Keith, 1970). The sensitivity of these labels to the local environment of the diamagnetic molecule is related to the geometry of the label itself, The two earliest structure reports in the literature, for di-*p*-anisyl nitroxide (Hanson, 1953) and di-*t*-butyl nitroxide (Andersen & Andersen, 1966), were consistent with a planar configuration at nitrogen but could not be regarded as completely conclusive as regards this point. Therefore, a report by Lajzérowicz-Bonneteau (1968) that a nonplanar nitroxide configuration existed in 2,2,6,6-tetramethyl-4-piperidinol-1-oxyl aroused considerable interest. This result was subsequently confirmed by Berliner (1970), although the angle between the N–O bond and the C–N–C plane was revised from the earlier value of 21° (Lajzérowicz-Bonneteau, 1968) to 15·8 ± 0·8°. At approximately the same time, the existence of planar forms of the nitroxide free radicals was definitively established by the remeasurement of the structure of potassium-2,2,5,5-tetramethyl-3-carboxypyrroline-1-oxyl (Boeyens & Kruger, 1970). We report here the crystal structure of a closely related compound, 2,2,5,5-tetramethyl-3-carbamidopyrroline-1-oxyl, wherein the configuration at nitrogen is again found to be planar, and where bond distances and angles are in excellent agreement with the values found by Boeyens & Kruger (1970).

This compound, $C_9H_{15}N_2O_2$, crystallizes in the monoclinic space group $P2_1/c$. The lattice constants ($a = 8{\cdot}039 \pm 0{\cdot}002$, $b = 11{\cdot}324 \pm 0{\cdot}002$, $c = 11{\cdot}537 \pm 0{\cdot}002$ Å, $\beta = 91{\cdot}75 \pm 0{\cdot}01°$), as determined by least-squares refinement of the setting angles for 10 reflections (Cu $K\alpha$ radiation) on a Picker four-circle goniostat, give a calculated density of 1·160 g.cm^{-3} for $Z = 4$. Of 1802 X-ray intensities measured on the diffractometer in 2θ-scan mode 1474 intensities that satisfied the condition $I > 3\sigma(I)$ were used for subsequent refinement. Absorption corrections were not needed ($\mu = 6{\cdot}85$ cm^{-1}), but a correction for secondary extinction was made. The phase problem was solved by iterative application of the $\sum_2$ formula (Dewar & Stone, 1968), and the positions of the nonhydrogen atoms were determined from an E map based on 300 reflections. All hydrogen atoms were subsequently located in a difference Fourier synthesis, and full-matrix least-squares refinement of atomic coordinates, hydrogen isotropic temperature factors, and C, N, and O anisotropic thermal parameters converged to a final conventional R value of 5·2 %. Scattering factors for C, N, and O were taken from *International Tables for X-Ray Crystallography* (1962), while those for hydrogen were from Stewart, Davidson & Simpson (1965). Table 1 shows the final atomic parameters. Structure factors are given in Table 2.

The structure is shown in Fig. 1, and bond distances and angles are given in Table 3. The N–O distance of 1·267 ± 0·005 Å, intermediate between an N–O double-bond length of 1·20 Å and a single-bond length of 1·44 Å (Pauling, 1960), is consistent with an electronic structure in which the bonding σ and π orbitals are filled by electron pairs, and the antibonding π^* orbital contains the lone electron. The N–O

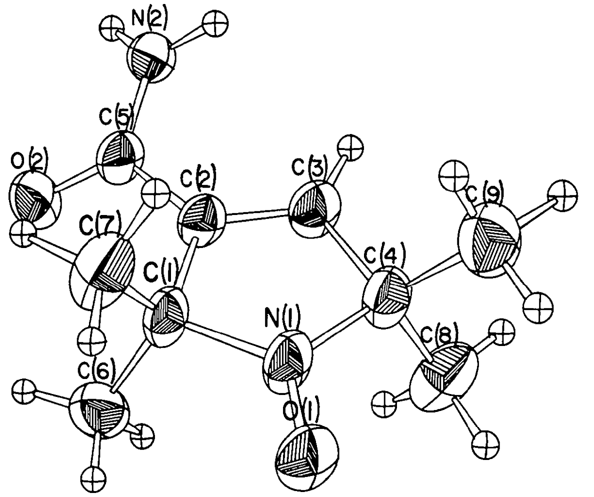

Fig. 1. Molecular structure of 2,2,5,5-tetramethyl-3-carbamidopyrroline-1-oxyl with thermal ellipsoids drawn at 50 % probability.

Table 1. *Final atomic parameters*

Anisotropic temperature factors are in the form: $\exp\,[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

	x	y	z	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
C(1)	0·7668 (3)	0·2748 (3)	0·2786 (2)	90 (4)	78 (3)	63 (2)	13 (3)	−5 (2)	17 (2)
C(2)	0·8028 (3)	0·3607 (2)	0·1823 (2)	108 (4)	60 (2)	52 (2)	7 (3)	−13 (2)	5 (2)
C(3)	0·9631 (3)	0·3676 (3)	0·1616 (2)	113 (4)	77 (3)	65 (2)	11 (3)	−3 (3)	21 (2)
C(4)	1·0684 (3)	0·2863 (3)	0·2339 (2)	96 (4)	88 (3)	66 (2)	10 (3)	0 (2)	20 (2)
C(5)	0·6682 (3)	0·4238 (2)	0·1154 (2)	103 (4)	63 (2)	57 (2)	5 (3)	−13 (2)	2 (2)
C(6)	0·6600 (5)	0·1685 (3)	0·2427 (4)	152 (6)	79 (3)	132 (4)	−18 (4)	−34 (4)	39 (3)
C(7)	0·6976 (5)	0·3359 (4)	0·3844 (3)	193 (7)	127 (4)	76 (3)	41 (5)	27 (4)	26 (3)
C(8)	1·1517 (5)	0·1913 (4)	0·1613 (3)	184 (7)	121 (4)	90 (3)	58 (5)	25 (4)	17 (3)
C(9)	1·1965 (5)	0·3499 (4)	0·3105 (4)	134 (6)	113 (4)	120 (4)	−8 (4)	−35 (4)	28 (4)
N(1)	0·9389 (3)	0·2331 (2)	0·3055 (2)	98 (4)	86 (2)	69 (2)	16 (2)	−8 (2)	30 (2)
N(2)	0·7086 (3)	0·5266 (2)	0·0684 (2)	109 (4)	66 (2)	76 (2)	−1 (2)	−31 (2)	13 (2)
O(1)	0·9710 (2)	0·1566 (2)	0·3834 (2)	147 (4)	112 (2)	91 (2)	25 (2)	−7 (2)	55 (2)
O(2)	0·5288 (2)	0·3799 (2)	0·1036 (2)	114 (3)	88 (2)	105 (2)	−14 (2)	−36 (2)	32 (2)

	x	y	z	B†(Å^2)		x	y	z	B(†Å^2)
H(1)	0·009 (4)	0·423 (3)	0·108 (3)	4·6 (0·7)	H(9)	0·697 (4)	0·277 (3)	0·445 (3)	6·6 (0·9)
H(2)	0·066 (6)	0·151 (4)	0·117 (4)	8·8 (1·4)	H(10)	0·582 (5)	0·372 (3)	0·364 (3)	7·5 (1·0)
H(3)	0·235 (5)	0·229 (3)	0·109 (3)	8·1 (1·1)	H(11)	0·665 (4)	0·110 (3)	0·302 (3)	6·2 (0·9)
H(4)	0·210 (4)	0·135 (3)	0·220 (3)	5·4 (0·8)	H(12)	0·542 (5)	0·188 (3)	0·228 (3)	8·1 (1·1)
H(5)	0·290 (4)	0·384 (3)	0·263 (3)	6·1 (0·9)	H(13)	0·713 (5)	0·127 (3)	0·174 (3)	6·9 (1·1)
H(6)	0·253 (4)	0·294 (3)	0·363 (3)	6·0 (0·8)	H(14)	0·635 (6)	0·561 (2)	0·017 (2)	3·3 (0·6)
H(7)	0·144 (5)	0·412 (4)	0·355 (4)	7·6 (1·2)	H(15)	0·810 (4)	0·560 (3)	0·083 (3)	4·4 (0·8)
H(8)	0·777 (5)	0·407 (4)	0·414 (3)	8·7 (1·2)					

† Isotropic temperature factor.

bond in nitroxides has also been described as a three-electron bond (Forrester, Hay & Thomson, 1968). The atoms C(1), C(4), N(1), and O(1) are coplanar within 0·004 Å, and the N–O bond forms an angle of less than 0·03° with the least-squares plane through the five ring atoms. (The maximum deviation of any atom from this plane is 0·013Å.)

Table 2. *Structure factors in* e × 100.

K	L	FOBS	FCALC
*** H = 0 ***			
0	0	0	0
2	0	7298	8476
4	0	4974	5067
6	0	2002	1989
8	0	1131	1161
10	0	1000	984
1	1	5386	6219
2	1	4688	4978
3	1	1021	1025
4	1	847	900
5	1	1800	1726
6	1	2132	2118
7	1	872	851
8	1	1010	1004
9	1	362	308
10	1	856	913
11	1	533	477
12	1	695	674
0	2	8232	9829
1	2	481	466
2	2	7527	8471
3	2	3464	3500
4	2	764	565
5	2	1091	1023
6	2	277	133
7	2	1941	1929
8	2	444	428
9	2	790	720
10	2	323	264
11	2	444	346
13	2	541	582
1	3	1303	1240
2	3	1958	1994
3	3	820	738
4	3	967	926
5	3	2592	2512
6	3	2171	2123
7	3	582	526
8	3	628	627
9	3	644	642
10	3	866	842
11	3	648	706
13	3	231	218
0	4	5403	5403
1	4	304	261
2	4	2085	1949
3	4	1996	1987
4	4	4700	4504
5	4	1250	1162
6	4	760	657
7	4	1763	1737
8	4	346	345
9	4	409	426
10	4	457	430
11	4	401	383
1	5	2801	2811
2	5	2095	2103
3	5	519	517
4	5	1563	1550
5	5	596	600
6	5	855	917
7	5	243	268
8	5	1175	1238
9	5	422	457
10	5	276	112
11	5	273	305
0	6	1431	1477
1	6	845	831
2	6	671	647
4	6	1027	980
5	6	2424	2393
7	6	889	890
8	6	743	763
10	6	279	330
11	6	294	296
1	7	390	317
3	7	727	710
4	7	995	928
5	7	1229	1310
6	7	1137	1246
7	7	461	464
8	7	1505	1593
11	7	240	126
0	8	433	420
1	8	1216	1160
2	8	388	325
3	8	766	793
4	8	1021	1047
5	8	358	322
6	8	281	29
7	8	701	692
8	8	975	981
9	8	255	161
10	8	305	254
2	9	598	616
3	9	358	331
4	9	459	371
5	9	292	249
6	9	1181	1244
7	9	882	914
8	9	542	602
9	9	214	54
0	10	877	897
1	10	674	701
2	10	285	270
3	10	291	245
4	10	452	414
5	10	1274	1279
6	10	438	391
9	10	273	276
2	11	431	447
3	11	374	399
4	11	422	444
5	11	256	92
6	11	368	353
0	12	276	75
1	12	360	300
3	12	681	710
4	12	304	136
5	12	312	281
6	12	326	301
2	13	449	454
*** H = 1 ***			
0	0	4998	5742
1	0	4605	4888
2	0	5674	6194
3	0	314	296
4	0	1300	1247
5	0	1830	1744
6	0	722	695
7	0	1476	1440
8	0	415	328
10	0	623	587
11	0	872	937
12	0	295	184
13	0	251	253
1	1	1565	1626
1	-1	3350	3397
2	-1	896	928
2	1	2108	2045
3	1	1465	1431
3	-1	2114	2170
4	-1	1079	1065
4	1	615	649
5	1	1090	1108
5	-1	1228	1159
6	-1	1064	1070
6	1	1553	1519
7	-1	936	883
8	-1	470	412
8	1	989	1007
9	-1	815	822
10	-1	281	170
10	1	621	616
11	1	569	565
12	-1	295	417
12	1	290	337
13	1	335	302
13	-1	250	216
0	-2	6389	7123
0	2	3499	3554
1	2	379	311
1	-2	4082	4195
2	-2	2763	2719
2	2	6685	7323
3	2	1314	1264
3	-2	662	636
4	-2	834	659
4	2	2400	2386
5	2	1248	1255
5	-2	728	715
6	-2	1129	1068
6	2	670	617
7	2	1084	1135
7	-2	702	711
8	-2	1316	1335
8	2	430	374
9	2	621	616
9	-2	907	905
10	-2	319	415
10	2	365	380
11	2	425	371
12	-2	559	558
12	2	347	383
1	3	3751	3782
1	-3	5851	6245
2	-3	8511	9582
2	3	1550	1510
3	3	960	974
3	-3	2751	2617
4	-3	2544	2461
4	3	2179	2098
5	3	285	268
5	-3	1102	1016
6	-3	1435	1447
6	3	351	430
7	3	638	577
7	-3	1071	1045
8	-3	654	672
8	3	296	257
9	3	856	861
9	-3	547	543
10	3	290	354
11	3	995	1073
11	-3	379	415
12	-3	248	234
0	-4	1303	1444
0	4	982	952
1	4	925	837
1	-4	1070	1052
2	-4	213	191
2	4	926	958
3	4	633	637
3	-4	4166	4142
4	-4	2587	2574
4	4	4732	4626
5	4	1228	1244
5	-4	2753	2662
6	-4	812	811
6	4	2913	2820
7	4	324	289
7	-4	323	221
8	-4	608	654
8	4	255	274
10	-4	261	65
10	4	1305	1383
11	4	586	516
12	4	479	455
1	5	1021	1070
1	-5	1404	1252
2	-5	2725	2657
2	5	242	170
3	5	2505	2433
3	-5	2615	2577
4	-5	1660	1781
4	5	215	156
5	5	977	935
5	-5	922	898
6	-5	537	513
6	5	1681	1625
7	5	320	382
7	-5	617	520
8	-5	938	1014
8	5	1402	1435
9	5	1657	1761
9	-5	659	610
10	5	602	605
11	5	841	876
12	-5	276	239
0	-6	2159	2199
0	6	3185	3176
1	6	1592	1527
1	-6	381	341
2	-6	835	750
2	6	734	521
3	6	1107	1115
3	-6	2313	2277
4	6	2760	2722
5	6	1256	1242
6	-6	1889	1858
6	6	2243	2202
7	-6	335	305
8	-6	768	744
8	6	1189	1278
9	6	746	740
9	-6	601	648
10	-6	227	53
10	6	820	877
11	6	332	246
11	-6	494	532
1	7	316	246
1	-7	691	670
2	-7	2878	2856
2	7	1700	1663
3	7	637	550
4	-7	560	569
5	-7	1321	1313
6	-7	783	747
6	7	2606	2667
7	7	1609	1700
7	-7	789	756
8	-7	617	561
8	7	743	722
9	7	232	209
9	-7	357	312
10	-7	407	461
10	7	354	330
0	-8	1384	1291
0	8	450	428
1	8	841	851
1	-8	1935	1858
2	-8	1085	1110
2	8	641	613
3	-8	1344	1283
4	8	413	378
5	8	1059	1079
5	-8	870	837
6	-8	903	847
6	8	585	608
7	8	812	786
7	-8	735	775
8	-8	673	657
8	8	253	155
9	8	296	280
1	9	734	723
2	-9	536	509
2	9	692	657
3	9	445	392
3	-9	376	333
4	-9	786	858
4	9	1854	1918
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5	7	484	513
6	7	557	617
6	-7	535	534
8	7	352	341
8	-7	317	286
9	-7	316	259
0	8	488	548
0	-8	1908	2006
1	8	1374	1409
2	8	489	468
2	-8	1183	1207
3	-8	349	318
3	8	536	534
4	8	1432	1449
4	-8	709	571
5	-8	462	492
5	8	337	270
7	8	277	233
1	9	320	312
2	9	778	786
2	-9	530	493
3	9	366	360
4	9	502	410
5	-9	351	259
5	9	599	618
6	9	228	128
6	-9	306	229
7	-9	287	300
0	10	900	927
0	-10	947	1013
1	-10	292	203
1	10	1122	1202
2	-10	368	310
3	-10	467	520
4	10	280	226
5	10	316	264
6	-10	231	253
1	-11	382	411
1	11	345	319
2	11	675	660
2	-11	347	336
4	-11	263	259
*** H = 6 ***			
0	0	1431	1414
1	0	984	1014
2	0	957	934
3	0	376	328
4	0	2036	2108
5	0	255	240
6	0	781	799
7	0	391	347
8	0	478	447
9	0	659	663
1	-1	1476	1439
2	-1	784	824
2	1	620	672
3	1	520	490
3	-1	710	685
4	-1	748	849
4	1	1624	1597
5	1	1307	1317
6	-1	723	764
6	1	964	963
8	-1	827	823
8	1	783	800
9	1	253	193
9	-1	332	236
10	-1	257	223
0	-2	1267	1270
0	2	321	298
1	2	1642	1647
2	-2	434	488
2	2	1036	1011
3	2	330	268
3	-2	631	605
4	2	335	284
4	-2	1769	1867
5	2	357	297
5	-2	744	773
6	-2	1352	1375
6	2	356	307
7	2	1143	1204
8	-2	313	308
9	-2	283	294
10	-2	284	320
1	3	1870	1848
1	-3	902	910
2	-3	349	354
2	3	2319	2318
3	3	272	183
3	-3	581	591
4	-3	550	440
4	3	751	716
5	3	778	900
5	-3	644	621
6	-3	1170	1242
6	3	624	631
7	3	911	885
7	-3	419	500
8	-3	951	966
8	3	266	141
0	-4	566	582
0	4	3308	3385
1	4	698	653
1	-4	863	890
2	-4	1207	1297
2	4	622	446
3	4	649	675
3	-4	650	647
4	-4	1259	1293
4	4	1284	1341
5	4	413	451
6	-4	1489	1521
6	4	608	608
7	4	435	423
8	-4	426	427
8	4	245	244
1	5	1259	1279
1	-5	380	288
2	5	1444	1439
3	-5	613	595
4	-5	384	342
6	-5	882	911
6	5	835	845
7	-5	551	516
8	5	416	393
0	-6	470	471
0	6	263	110
1	6	528	489
1	-6	1179	1155
2	-6	762	796
3	6	1001	1018
3	-6	559	552
4	-6	1171	1213
4	6	1150	1169
5	6	484	567
5	-6	768	797
6	6	335	268
7	6	785	825
7	-6	380	374
8	6	233	258
1	7	1034	1039
1	-7	569	567
2	-7	367	285
3	7	1031	1133
3	-7	877	901
4	-7	900	920
4	7	476	482
5	7	540	556
5	-7	258	141
6	-7	303	318
6	7	575	570
7	7	564	570
7	-7	490	483
0	-8	709	762
0	8	1917	1921
1	8	414	362
1	-8	421	492
2	-8	521	506
2	8	438	329
3	-8	292	208
4	8	335	326
5	8	314	277
5	-8	415	425
6	-8	323	206
7	-8	362	341
1	9	634	585
1	-9	266	214
2	-9	527	546
2	9	215	126
3	-9	281	222
4	9	723	734
0	-10	590	555
0	10	429	405
1	-10	347	241
2	-10	215	94
2	10	699	745
*** H = 7 ***			
0	0	1333	1410
1	0	1439	1463
2	0	1348	1383
3	0	1262	1311
5	0	1165	1196
6	0	674	707
7	0	308	229
8	0	283	289
1	-1	521	487
1	1	571	546
2	1	2170	2246
2	-1	540	459
3	-1	459	506
3	1	809	801
4	1	418	385
4	-1	272	253
5	-1	1017	1064
5	1	428	409
6	1	450	390
6	-1	806	810
7	1	415	336
0	2	2974	3090
0	-2	924	879
2	2	495	284
2	-2	1514	1544
3	-2	1073	1146
3	2	605	610
4	2	480	408
4	-2	1336	1415
5	-2	552	505
6	-2	557	493
7	-2	303	262
8	2	623	625
8	-2	277	259
1	-3	323	264
1	3	1377	1341
2	3	1337	1298
2	-3	1428	1462
3	3	684	669
4	3	758	797
4	-3	372	364
5	-3	317	248
5	3	230	79
6	3	579	610
6	-3	459	431
7	3	415	355
8	3	353	345
8	-3	345	283
0	4	1367	1424
0	-4	742	691
1	-4	1810	1795
1	4	487	432
2	4	1206	1233
2	-4	736	713
3	-4	241	214
3	4	917	894
4	4	315	237
4	-4	947	926
5	-4	304	156
5	4	775	877
6	4	941	935
7	4	547	576
8	-4	416	408
1	-5	379	330
1	5	511	445
2	5	614	610
3	-5	509	557
3	5	427	411
4	5	248	217
4	-5	588	522
5	-5	434	375
5	5	471	492
6	5	838	870
7	-5	214	60
7	5	266	284
0	-6	261	240
1	-6	549	545
1	6	621	657
2	6	1001	982
2	-6	283	40
3	-6	321	192
5	6	307	236
6	6	548	555
6	-6	675	680
7	-6	392	427
1	7	678	654
2	7	434	436
3	7	545	560
4	-7	441	436
5	-7	309	363
5	7	253	230
0	8	394	376
1	-8	404	440
1	8	237	192
2	8	575	550
2	-8	371	345
3	-8	397	449
3	8	315	259
*** H = 8 ***			
0	0	1671	1659
1	0	465	455
2	0	471	396
3	0	309	267
5	0	542	581
1	-1	303	246
2	-1	917	953
2	1	412	377
3	1	347	250
3	-1	227	56
4	-1	304	282
4	1	877	910
5	1	457	431
5	-1	484	464
6	-1	404	362
6	1	381	330
7	1	264	272
7	-1	209	114
0	-2	1614	1589
1	2	249	132
1	-2	409	367
2	-2	817	815
2	2	315	314
3	-2	574	600
4	-2	857	917
4	2	703	688
5	2	248	174
1	3	250	220
1	-3	505	505
2	-3	307	186
3	3	604	622
3	-3	868	902
4	-3	462	477
4	3	345	311
5	3	507	500
5	-3	288	307
6	-3	229	194
0	-4	241	198
1	4	333	249
1	-4	317	188
2	4	543	572
3	4	267	206
3	-4	533	521
4	-4	472	431
4	4	335	282
5	4	591	564
5	-4	421	325
6	-4	316	341
1	5	467	449
1	-5	340	275
2	-5	709	768
3	5	728	724
3	-5	338	289
4	5	437	367
5	-5	325	333
0	-6	878	903
0	6	699	760
1	6	504	474
2	-6	302	216
2	6	442	404
3	6	421	394
3	-6	510	479
4	-6	217	190
1	-7	274	290
2	-7	229	168
*** H = 9 ***			
0	0	271	223
1	0	757	735
3	0	272	211
4	0	451	440
1	-1	225	130
2	1	460	456
2	-1	612	598
3	-1	293	232
3	1	371	441
0	2	292	230
0	-2	288	189
1	-2	273	164
1	2	438	420
2	2	364	294
2	-2	278	219
3	-2	623	607
1	3	287	270
2	3	484	468
3	-3	331	323
0	-4	254	186
1	-4	302	276

Table 3. *Bond distances and angles*

Selected distances (Å)*	
C(1)–C(2)	1·511 (7)
C(2)–C(3)	1·319 (7)
C(3)–C(4)	1·487 (7)
C(4)–N(1)	1·477 (7)
N(1)–C(1)	1·486 (6)
N(1)–O(1)	1·267 (5)
C(2)–C(5)	1·490 (7)
C(5)–O(2)	1·230 (7)
C(5)–N(2)	1·329 (7)
C(1)–C(6)	1·526 (8)
C(1)–C(7)	1·521 (8)
C(4)–C(8)	1·529 (9)
C(4)–C(9)	1·517 (8)
O(1)–N(2)	3·003 (6)
O(1)–H(15)	2·097
N(2)–H(15)	0·883
O(2)–N(2)	2·908 (6)
O(2)–H(14)	2·000
N(2)–H(14)	0·898

Angles (°)			
N(1)–C(1)–C(2)	99·2	N(1)–C(4)–C(3)	99·7
N(1)–C(1)–C(6)	108·5	N(1)–C(4)–C(8)	110·2
N(1)–C(1)–C(7)	109·7	N(7)–C(4)–C(9)	110·1
C(2)–C(1)–C(6)	115·3	C(3)–C(4)–C(8)	112·4
C(2)–C(1)–C(7)	112·2	C(3)–C(4)–C(9)	113·3
C(6)–C(1)–C(7)	111·2	C(8)–C(4)–C(9)	110·7
C(1)–C(2)–C(3)	112·5	C(1)–N(1)–C(4)	114·8
C(1)–C(2)–C(5)	122·4	C(1)–N(1)–O(1)	122·2
C(3)–C(2)–C(5)	125·1	C(4)–N(1)–O(1)	123·0
C(2)–C(3)–C(4)	113·8	C(2)–C(5)–N(2)	116·6
H(1)–C(3)–C(4)	122·4	C(2)–C(5)–O(2)	120·5
H(1)–C(3)–C(2)	123·7	N(2)–C(5)–O(2)	122·8

* Errors in the last significant figure are given in parentheses for distances, and are approximately 0·5° for angles involving nonhydrogen atoms.

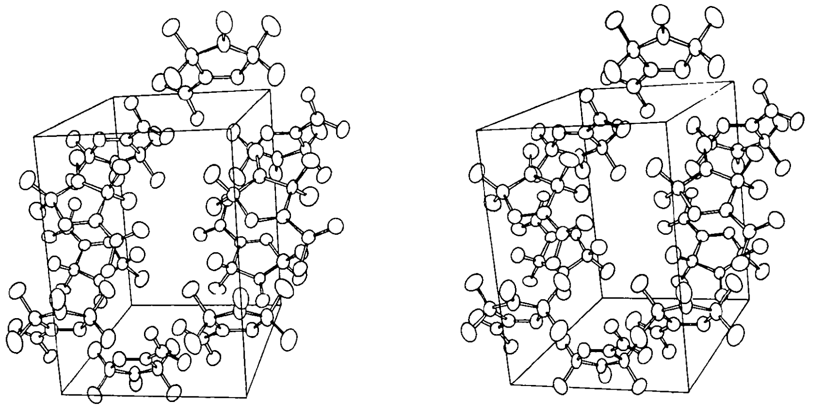

Fig. 2. Crystal structure of 2,2,5,5-tetramethyl-3-carbamidopyrroline-1-oxyl. The *b* axis is vertical and the *a* axis horizontal.

The heterocyclic C–N distances of 1·477 ± 0·007 and 1·486 ± 0·006 Å, and the C(2)=C(3) distance of 1·319 ± 0·007 Å are normal (Sutton, 1965) and indicate the absence of electron delocalization in the ring. The plane of the amide group makes an angle of 29·0° with that of the ring.

Two types of N–H···O hydrogen bonds, both linear and fairly weak, exist in the crystal structure (Fig. 2). The shorter type, of length 2·908 Å, occurs in pairs between the amide nitrogen atoms and the carbonyl oxygen atoms of adjacent molecules to form dimers across centers of symmetry. Even weaker interactions, of length 3·003 Å, link the amide groups to the oxyl oxygen atoms of other molecules to form a three-dimensional network.

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